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Technical Report No. T17

$\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, a New Berylloarsenate Phase Containing "Bridged" Tetrahedral 3-Rings

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**$\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, a New Beryllioarsenate Phase
Containing "Bridged" Tetrahedral 3-Rings**

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ABSTRACT

The high-temperature/high-pressure hydrothermal synthesis and X-ray single crystal structure of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ is described. The title compound contains a three-dimensional network of BeO_4 and AsO_4 tetrahedra. The structural motif includes infinite layers of "bridged" tetrahedral 3- and 4-rings. ^9Be MAS NMR data are consistent with the Be-atom environments in the crystal structure.

Crystal data: $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ ($M_r = 170.45$), monoclinic, space group $C2/c$, $a = 16.318(2) \text{ \AA}$, $b = 4.6664(3) \text{ \AA}$, $c = 9.8755(7) \text{ \AA}$, $\beta = 93.777(3)^\circ$, $V = 750.37 \text{ \AA}^3$, $Z = 4$. Final agreement values of $R = 3.59\%$ and $R_w = 4.44\%$ were obtained for 1289 observed reflections with $I > 3\sigma(I)$.

Introduction

Molecular sieves containing tetrahedral 3-rings as part of their structures are currently of interest (1). It has been suggested (2) that phases containing 3-rings as part of their three-dimensional structure may have a particularly-low tetrahedral-framework-atom density. The new aluminosilicate ZSM-18 (3) contains 3-rings of T -atoms ($T = \text{Si}, \text{Al}$) and is notable as a large-pore material containing 1-dimensional channels, which may have technologically-valuable properties. The novel microporous beryllsilicate phase lovdarite (4) also contains 3-rings, as does a zincosilicate phase (5) which contains a framework sub-unit built up from three TO_4 units. Many possible topologies of 3-ring-containing structures have been recently elucidated (6).

We have previously described several systems containing various types of tetrahedral 3-rings in the group 2/12/15 (beryllo/zinco)(phosphate/arsenate) phase space (7). The one-dimensional chain-ions found in $\text{Na}_2\text{ZnPO}_4\text{OH}\cdot 7\text{H}_2\text{O}$ and synthetic bearsite, $\text{Be}_2\text{AsO}_4\text{OH}\cdot 4\text{H}_2\text{O}$ contain tetrahedral 3-rings as part of their structure (8). The two-dimensional structures of $\text{CsH}(\text{ZnPO}_4)_2$ and $\text{NaH}(\text{ZnPO}_4)_2$ contain "bridged" 3-rings as part of anionic layers sandwiching cesium and sodium cations (9). Finally, the novel three-dimensional structure of $\text{Zn}_3(\text{PO}_4)(\text{HPO}_4)_2\cdot \text{HN}_2\text{C}_6\text{H}_{12}$ contains similar "bridged" 3-rings, as part of its open, 8-ring-channel containing framework (10). Here, the term "bridged" 3-ring refers to the fact that one of the oxygen-atom $T\text{-O-T'}$ bridges in the tetrahedral 3-ring is bonded to three neighboring tetrahedral atoms, compared to the usual two T -atom neighbors for other $T\text{-O-T'}$ bridges (Figure 1).

In this paper we describe the synthesis and characterization of $\text{Be}_3(\text{AsO}_4)_2\cdot 2\text{H}_2\text{O}$, a new beryllium arsenate hydrate phase, which also contains "bridged" 3-rings of BeO_4 and AsO_4 groups as part of its three-dimensional structure.

Synthesis and Structural Characterization

The title compound was prepared by a high-temperature/high-pressure hydrothermal reaction: 0.087 g of $\text{Be}(\text{OH})_2$, 0.3 ml of H_2O and 0.644 g of 4M H_3AsO_4 solution were sealed in a gold tube and heated to 550°C for 135 hours in a Leco TEMPRESTM bomb. Upon cooling, numerous needle- and shard-like crystals were recovered from the mother liquor ($\text{pH} = 2$) by vacuum filtration. *Warning!*: beryllium and arsenic compounds are extremely toxic. Take all normal safety precautions in handling these materials.

The structure of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ was determined by standard single-crystal X-ray methods (11). A suitable shard of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ was selected and mounted on a thin glass fiber with cyanoacrylate adhesive. As-synthesized transparent $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ crystals are shard- or needle-like and have a maximum linear dimension of ~ 0.5 mm.

Room temperature [$25(2)^\circ\text{C}$] intensity data were collected on a Huber automated 4-circle diffractometer (graphite-monochromated $\text{Mo K}\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$). After locating and centering of 25 reflections, unit cell constants were optimized by least-squares refinement, resulting in monoclinic lattice parameters of $a = 16.318(2) \text{ \AA}$, $b = 4.6664(3) \text{ \AA}$, $c = 9.8755(7) \text{ \AA}$, $\beta = 93.777(3)^\circ$ (e.s.d.s in parentheses). Intensity data were collected in the θ - 2θ scanning mode with standard reflections monitored for intensity variation throughout the course of each experiment. The scan speed was $6^\circ/\text{min}$ with a scan range of 1.3° below $\text{K}\alpha_1$ to 1.6° above $\text{K}\alpha_2$. No significant variation in standards was observed. The raw data were reduced using a Lehmann-Larsen profile-fitting routine (12) and the normal corrections for Lorentz and polarization effects were made. The systematic absences in the reduced data (hkl , $h+k$; $h0l$, l) indicated space group $C2/c$ (No. 15).

The crystal structure of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ was partially solved using the direct-methods program SHELXS-86 (13), and other atom positions, including proton sites, were located

from Fourier difference maps during the refinement procedure. After isotropic refinement, an empirical absorption correction (DIFABS) (14) was applied (minimum correction = 0.9, max. = 1.6). The final cycles of full-matrix least-squares refinement were against F and included anisotropic temperature factors (atom-type isotropic thermal factors for the protons) and a secondary extinction correction (15). Complex, neutral-atom scattering factors were obtained from *International Tables* (16).

Hydrogen-atom positions were refined with bond distance [$d(\text{O-H}) = 0.95(1) \text{ \AA}$] and bond angle [$\theta(\text{H-O-H}) = 109(1)^\circ$] restraints. At the end of the refinement, analysis of the various trends in F_o versus F_c revealed no unusual effects. The least-squares, Fourier and subsidiary calculations were performed using the Oxford CRYSTALS system (17), running on a DEC MicroVAX 3100 computer. Supplementary tables of observed and calculated structure factors and anisotropic thermal factors are available from the authors. Crystallographic data are summarized in Table I.

^9Be MAS NMR data for the Be/As/O sample were collected on a Brücker/Nicolet NT-200 hybrid spectrometer system, at 28.12 MHz (field strength 4.1 T) with 785 acquisitions, using a 7 mm broadband MAS NMR probe from Doty Scientific, spinning at a speed of approximately 5 KHz. Data were collected in a single-pulse mode, with a 45° pulse length of $3 \mu\text{s}$ and a recycle delay time of 60 seconds. A broad singlet at -1.38 ppm is evident, as referenced to 2M $\text{Be}(\text{NO}_3)_2$ solution (downfield shifts positive).

Results

Final atomic positional and thermal parameters for $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ are listed in Table II, with selected bond distance/angle data in Tables III and IV respectively. The asymmetric unit and atom-labeling scheme are illustrated in Figure 2. $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ is built up

from BeO_4 and AsO_4 subunits, as illustrated in Figure 3 in polyhedral representation (18).

This connectivity of tetrahedral nodes in $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ leads to a three-dimensional network, built up from infinite *bc*-plane layers of As(1) and Be(2) tetrahedral groups, which are linked together by the Be(1) O_4 groups. The As(1)/Be(2) layers (Figure 4) are built up from infinite tetrahedral "bridged" 3- and 4-rings. The "backbone" of the layer is an edge-sharing chain of 4-rings, each of configuration $\overline{\text{Be}(2) - \text{As}(1) - \text{Be}(2) - \text{As}(1)}$, which propagates in the *b*-direction. Adjacent 4-ring chains are fused together by 3-rings (configuration $\overline{\text{Be}(2) - \text{As}(1) - \text{Be}(2)}$), which are fused edge-to-edge themselves, resulting in an infinite sheet structure. A notable feature of this connectivity is the fact that each Be(2) atom makes contact with six nearest-neighbor tetrahedral atoms [four distinct As(1)s and two distinct Be(2)s], and each As(1) atom bonds to six different Be(2) atoms, rather than the usual four nearest neighbor *T*-atoms as found in zeolite molecular sieves.

Be(1) has two water-molecule oxygen atoms [atom O(1)] in its coordination sphere, and also makes two bonds to two different As atoms via O(3), with $d_{\text{av}}(\text{Be}-\text{O}) = 1.633(4) \text{ \AA}$. Be(2) makes four distinct bonds to As via O(2), O(4), O(4)' and O(5) [$d_{\text{av}}(\text{Be}-\text{O}) = 1.635(3) \text{ \AA}$]. The tetrahedrally-coordinated As(1) atom [$d_{\text{av}}(\text{As}-\text{O}) = 1.683(2) \text{ \AA}$] makes four As-O-Be bonds; an As(1)-O(3)-Be(1) bond, and links to Be(2) via O(2), O(4) and O(5). The bridging oxygen atoms fall into two groups; O(2), O(3) and O(5) link adjacent Be and As species, as noted above, with $\theta_{\text{av}}(\text{Be}-\text{O}-\text{As}) = 127(1)^\circ$. O(4) bonds to three nearest neighbor tetrahedral species; $2 \times \text{Be}(2)$ and As(1), with the geometrical parameters listed in Tables III and IV. Of the two protons attached to O(1), H(2) is involved in an H-bonding contact to O(2) [$d(\text{O} \cdots \text{H}) = 1.80(1) \text{ \AA}$], while H(1) has no near neighbors.

Brown/Wu bond-valence-sum (BVS) values (19) accord with these bonding geometries; Be(1) has a BVS of 2.02, Be(2) 2.00 and As(1) 5.02 (expected values for Be and As = 2.00

and 5.00 respectively). The values for the O-atom species are: O(1), neglecting protons 0.45; O(2) 1.81; O(3) 1.88; O(4) 2.02; O(5) 1.88 (expected values 2.00). In this phase it therefore appears that the three-coordinated oxygen atom has a "typical" BVS value, while the oxygen atoms which bridge two tetrahedral neighbors are slightly "underbonded." This situation is reversed in the layered phases $M\text{H}(\text{ZnPO}_4)_2$ ($M = \text{Cs}, \text{Na}$) (9) where the bridgehead (3-coordinate) oxygen atom appears to be somewhat "overbonded," with a BVS of ~ 2.2 , compared to the typical value of ~ 2 for the other O-atoms.

The ^9Be MAS NMR spectrum of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ (Figure 5) indicates that only one magnetically-distinct Be atom site is observable at this resolution, even though there are two crystallographically-distinct Be-atom sites in the structure. The chemical shift observed is in agreement with previous studies (20, 21). The observation of a single resonance may be rationalized by considering the consistent singular local environment (orthoberyl-ate group, BeO_4) of the Be-atoms throughout the crystal, regardless of the fact that the next-nearest-neighbors are not equivalent. Similar instances have been documented in one- two- and three-dimensional systems containing BeO_4 units (8, 20) and also by $^{31}\text{P}/^9\text{Be}$ MAS NMR studies of other beryllophosphate phases (21).

Discussion

Rather similar sheet configurations to the three-/four-ring layers found in $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$ have been found in the layered, anionic zincophosphate phases $\text{CsH}(\text{ZnPO}_4)_2$ and $\text{NaH}(\text{ZnPO}_4)_2$ (9). However, the precise nature of the tetrahedral-atom connectivity is different in each case: In $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, the 3-rings are fused with each other as described above. In $\text{CsH}(\text{ZnPO}_4)_2$, the tetrahedral 3-rings are joined "head to tail" and no two 3-rings share a common oxygen atom, while in $\text{NaH}(\text{ZnPO}_4)_2$ the 3-rings are isolated (from each other) and interlinked by tetrahedral 4-rings.

Conversely, the one-dimensional phases $\text{Na}_2\text{ZnPO}_4\text{OH}\cdot 7\text{H}_2\text{O}$ and $\text{Be}_2\text{AsO}_4\text{OH}\cdot 4\text{H}_2\text{O}$ contain "true" (unbridged) tetrahedral 3-rings as part of their chain structures (8). In these phases, hydroxide-ion bridges (i.e., $\text{Zn}-(\text{OH})-\text{Zn}$ and $\text{Be}-(\text{OH})-\text{Be}$ in $\text{Na}_2\text{ZnPO}_4\text{OH}\cdot 7\text{H}_2\text{O}$ and $\text{Be}_2\text{AsO}_4\text{OH}\cdot 4\text{H}_2\text{O}$ respectively) bonds replace the bridgehead three-coordinate O-atom found in $\text{Be}_3(\text{AsO}_4)_2\cdot 2\text{H}_2\text{O}$.

In conclusion, the synthesis and structure of $\text{Be}_3(\text{AsO}_4)_2\cdot 2\text{H}_2\text{O}$, a new beryllium arsenate hydrate phase, have been described. Like several other phases in the $(\text{Be}/\text{Zn})(\text{P}/\text{As})\text{O}$ -structure field, this material contains tetrahedral 3-rings, although they are bridged or "fused," by way of a 3-coordinate oxygen atom which links the 3-ring group with another tetrahedral-atom center. By way of contrast, such tri-bridging oxygen atoms appear to be unknown in tetrahedral aluminosilicate structures. The non 1:1 Be:As ratio in $\text{Be}_3(\text{AsO}_4)_2\cdot 2\text{H}_2\text{O}$ leads to a neutral "framework," as opposed to the typical anionic framework found in other BeAsO sieves (7), which suggests the possibility of synthesizing other three-dimensional, neutral BeAsO materials containing 3-rings. Efforts are now being made to that effect.

Acknowledgement We thank the National Science Foundation (Grant #DMR-9208511) and the Office of Naval Research for partial financial support.

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Figure Captions

Figure 1: Partial view of tetrahedral-Zn/P 3-rings linked via a "bridgehead" oxygen atom, O(1) in the three-dimensional phase $\text{Zn}_3(\text{PO}_4)(\text{HPO}_4)_2 \cdot \text{HN}_2\text{C}_6\text{H}_{12}$ (see text and ref. 10).

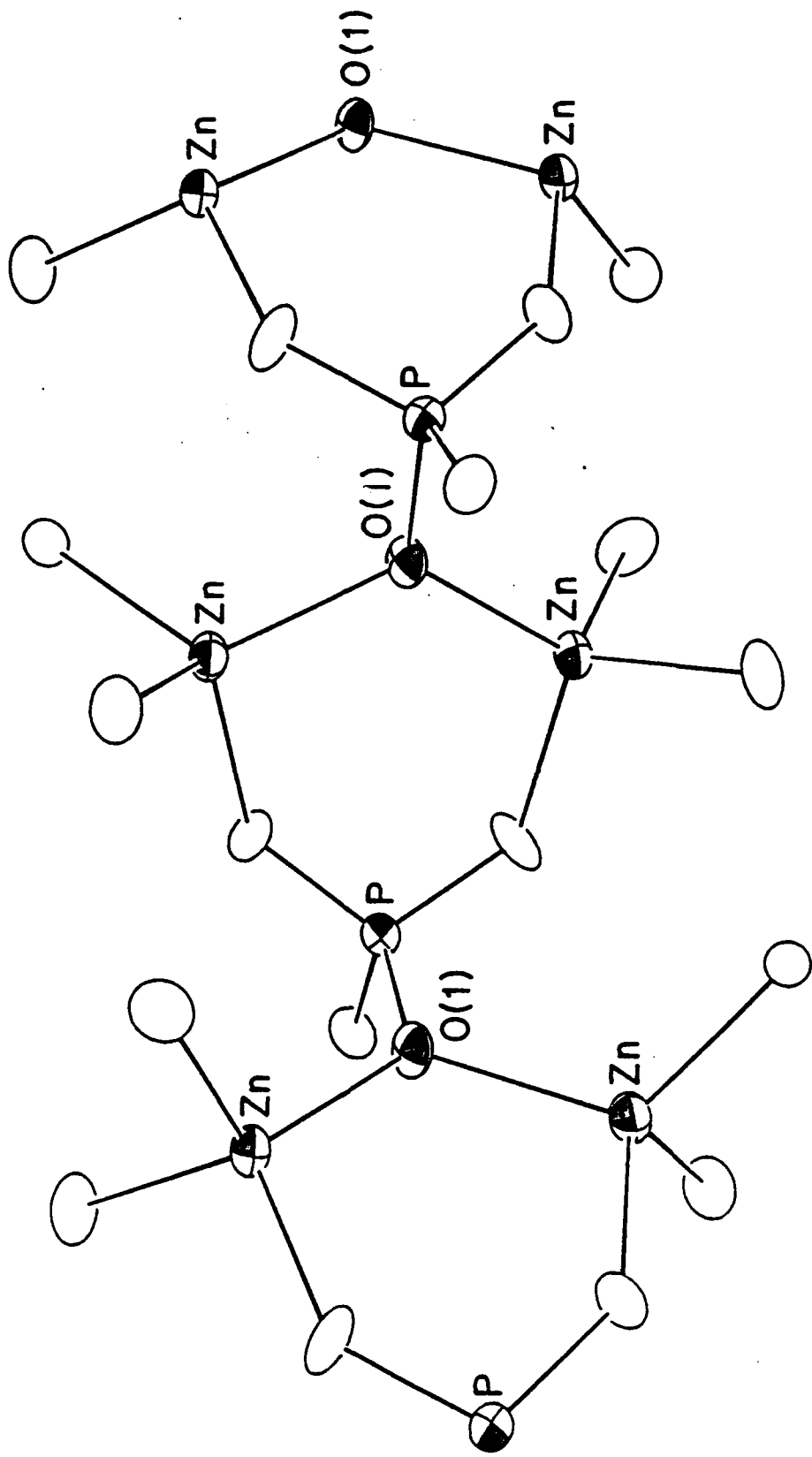
Figure 2: Asymmetric unit of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, showing the atom-labeling scheme.

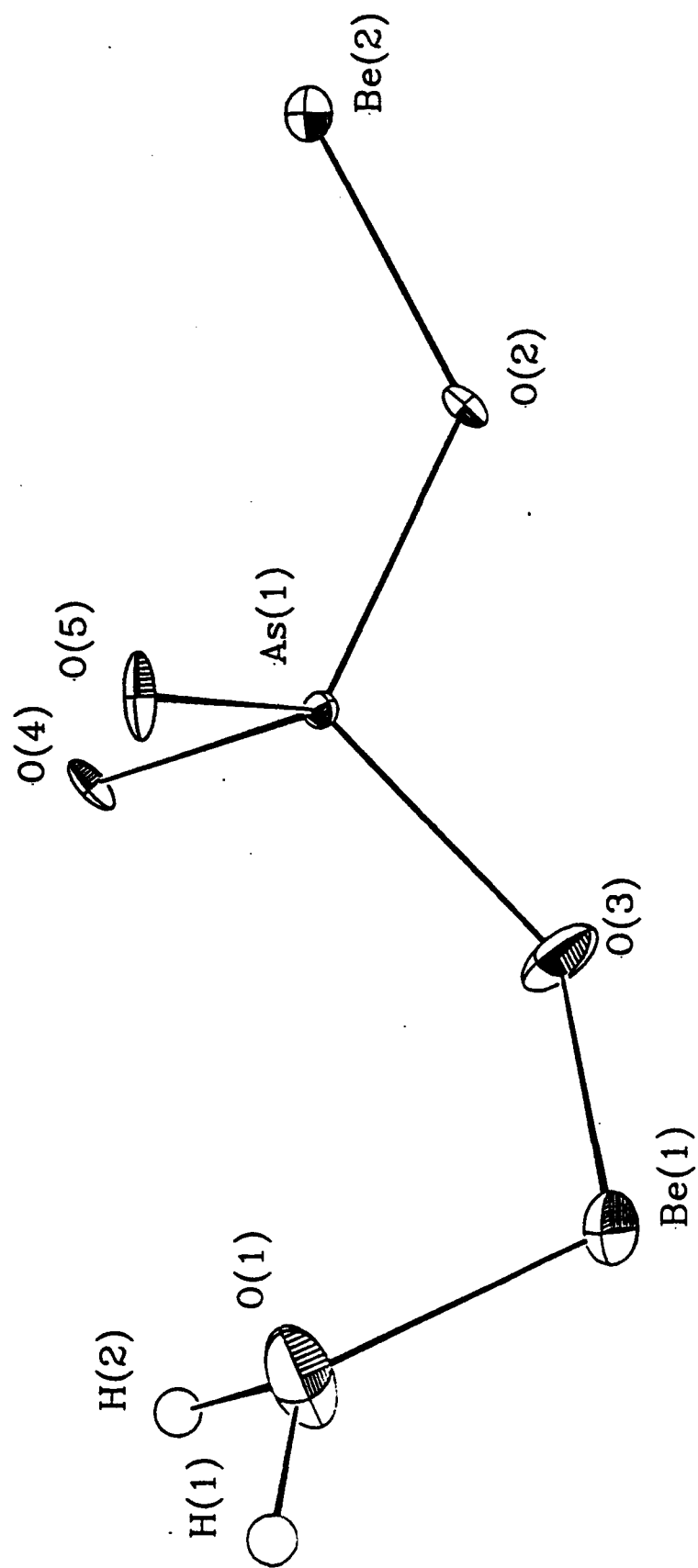
Figure 3: Polyhedral plot of the crystal structure of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, viewed down the b-direction. The "hanging" tetrahedral vertices of the interlayer $\text{Be}(1)\text{O}_4$ groups are occupied by water molecules (see text).

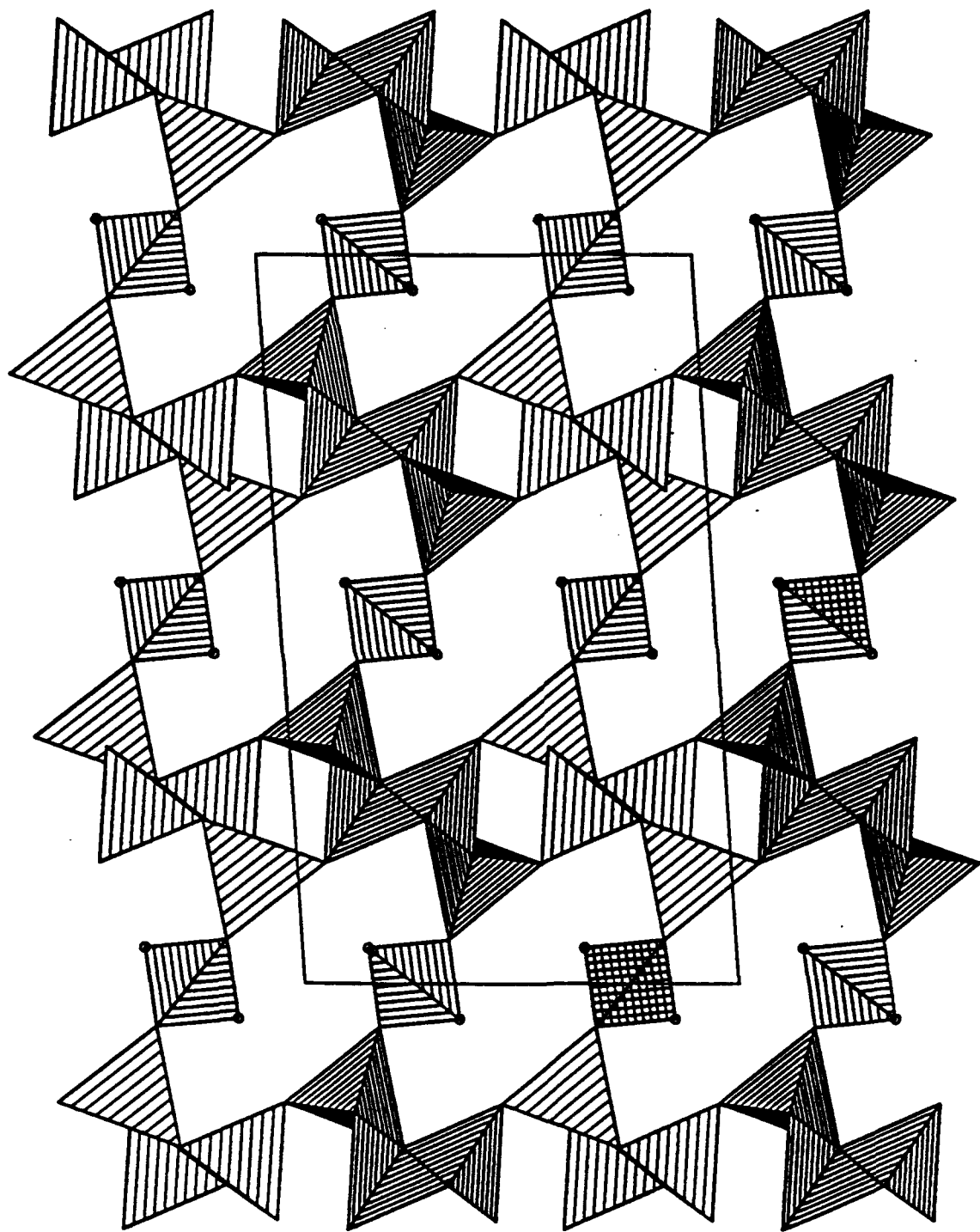
Figure 4: ORTEP view of the $\text{Be}(2)/\text{As}(1)$ -centered tetrahedral layers in the bc-plane of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, showing the interlinked 3- and 4-rings of tetrahedra.

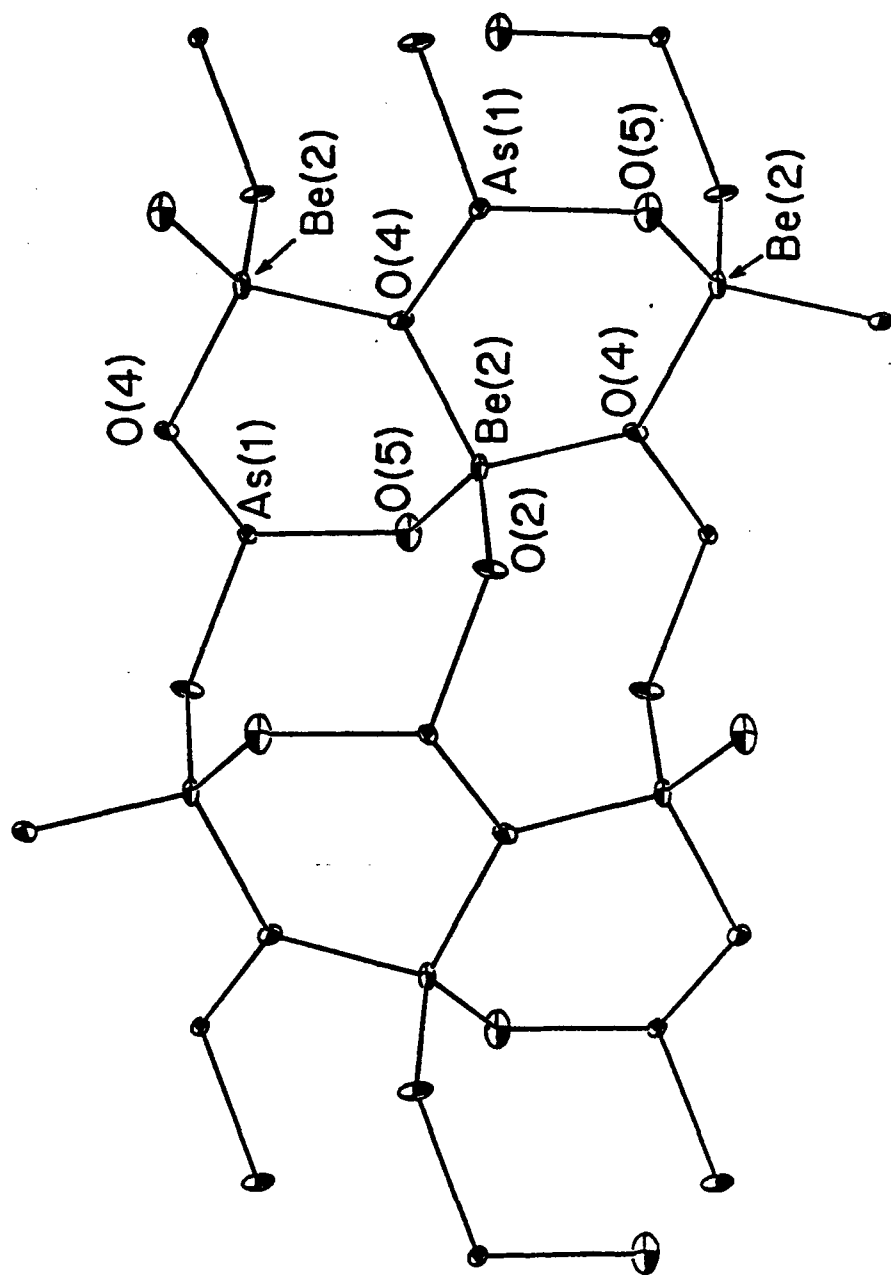
Figure 5: ^9Be MAS NMR spectrum of $\text{Be}_3(\text{AsO}_4)_2 \cdot 2\text{H}_2\text{O}$, referenced to 2M $\text{Be}(\text{NO}_3)_2$.

Fig. 1.









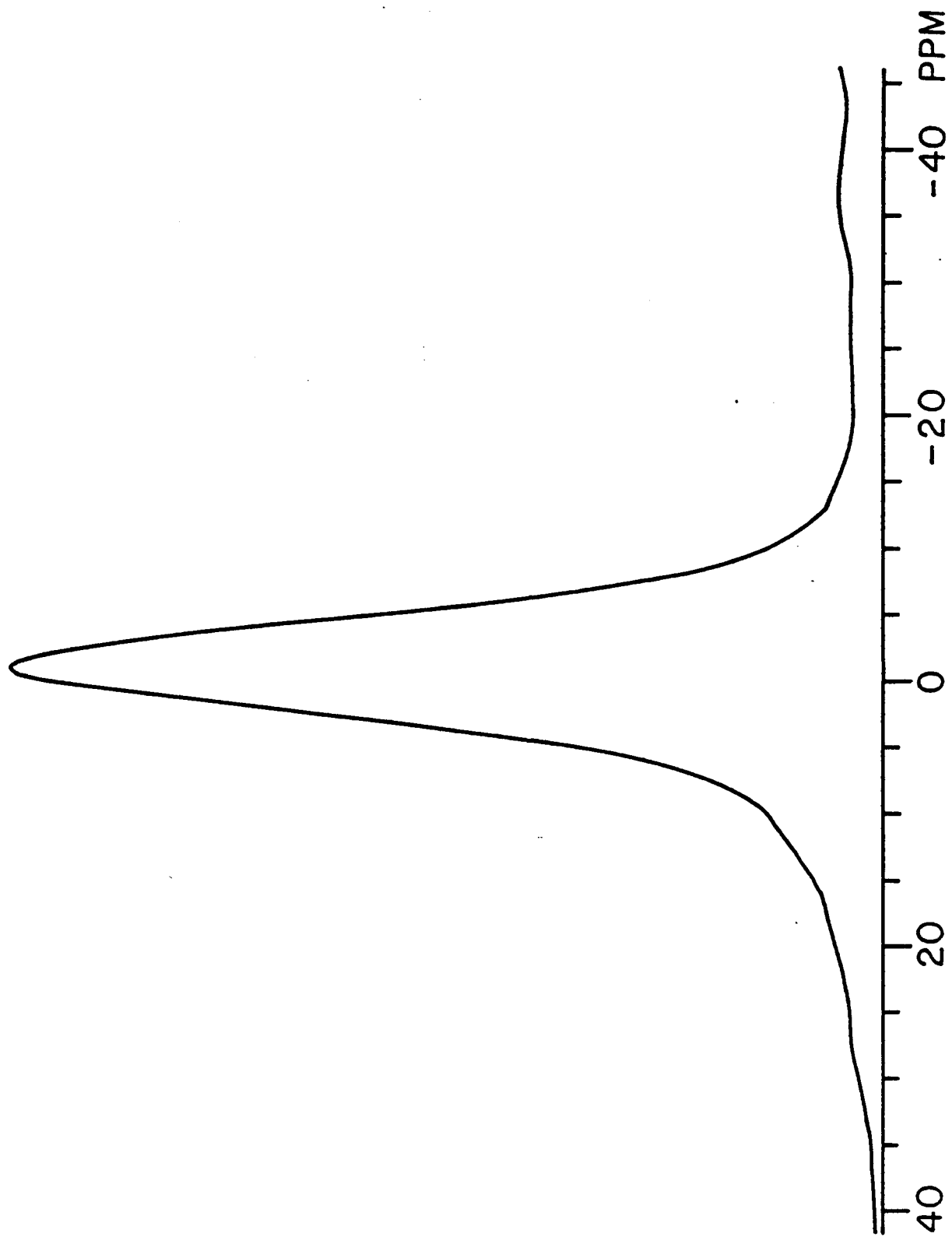


Table I: Crystallographic Parameters

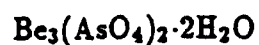


empirical formula	$\text{As}_2\text{O}_{10}\text{Be}_3\text{H}_4$
formula wt.	170.45
habit	colorless shard
crystal system	monoclinic
a (Å)	16.318 (2)
b (Å)	4.6664 (3)
c (Å)	9.8755 (7)
β (°)	93.777 (3)
V (Å ³)	750.37
Z	4
space group	$C2/c$ (No. 15)
T (°C)	25 (1)
$\lambda(\text{Mo K}\alpha)$ (Å)	0.71073
ρ_{calc} (g/cm ³)	3.02
$\mu(\text{Mo K}\alpha)$ (cm ⁻¹)	89.2
absorption correction	DIFABS
hkl limits	$\pm 24, +7, +14$
total data	1609
observed data†	1289
$R(F_o)^a$ (%)	3.59
$R_w(F_o)^b$ (%)	4.44

† $I > 3\sigma(I)$

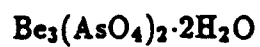
$$R^a = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|, R_w^b = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w|F_o|^2]^{1/2}$$

Table II: Atomic Positional Parameters



Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
Be(1)§	1/2	0.195 (2)	1/4	0.0098
Be(2)	0.2528 (3)	-0.056 (1)	-0.1606 (5)	0.0048
As(1)	0.34532 (2)	0.05276 (8)	0.09835 (4)	0.0034
O(1)	0.4526 (2)	0.4238 (8)	0.3494 (4)	0.0151
O(2)	0.3350 (2)	-0.0772 (7)	-0.0597 (3)	0.0057
O(3)	0.4399 (2)	-0.0159 (8)	0.1646 (4)	0.0103
O(4)	0.2764 (2)	-0.1100 (7)	0.1984 (3)	0.0046
O(5)	0.3237 (2)	0.4018 (7)	0.0993 (3)	0.0060
H(1)	0.474 (8)	0.58 (3)	0.40 (1)	0.05 (2)†
H(2)	0.419 (7)	0.33 (3)	0.41 (1)	0.05 (2)†

§Wyckoff site 4e (symmetry 2), $^*U_{eq}(\text{\AA}^2) = (U_1 U_2 U_3)^{1/3}$, † U_{iso}

Table III: Bond Distances (Å)

Be(1)–O(1)	1.674(7)	Be(1)–O(1)'	1.674(7)
Be(1)–O(3)	1.591(6)	Be(1)–O(3)'	1.591(6)
Be(2)–O(2)	1.621(6)	Be(2)–O(4)	1.667(6)
Be(2)–O(4)	1.660(6)	Be(2)–O(5)	1.593(6)
As(1)–O(2)	1.673(3)	As(1)–O(3)	1.668(3)
As(1)–O(4)	1.722(3)	As(1)–O(5)	1.667(3)
O(1)–H(1)	0.9(1)	O(1)–H(2)	0.9(1)
O(2)···H(2)	1.8(1)†		

† H-bond contact

Table IV: Bond Angles (°)



O(1)-Be(1)-O(1)'	100.7(5)	O(3)-Be(1)-O(1)	114.4(2)
O(3)-Be(1)-O(1)'	112.1(2)	O(3')-Be(1)-O(1)	112.1(2)
O(3')-Be(1)-O(1)'	114.4(2)	O(3)-Be(1)-O(3)'	103.6(5)
O-Be(1)-O	109.6[5.9]†		
O(4)-Be(2)-O(2)	107.0(3)	O(4)-Be(2)-O(2)	108.5(3)
O(4)-Be(2)-O(4)	109.0(3)	O(5)-Be(2)-O(2)	115.6(3)
O(5)-Be(2)-O(4)	106.7(3)	O(5)-Be(2)-O(4)	109.8(3)
O-Be(2)-O	109.4[3.2]†		
O(3)-As(1)-O(2)	109.3(2)	O(4)-As(1)-O(2)	110.3(1)
O(4)-As(1)-O(3)	108.2(2)	O(5)-As(1)-O(2)	110.6(2)
O(5)-As(1)-O(3)	112.1(2)	O(5)-As(1)-O(4)	106.3(1)
O-As(1)-O	109.5[2.0]†		
As(1)-O(2)-Be(2)	125.4(3)	As(1)-O(3)-Be(1)	127.7(3)
Be(2)-O(4)-Be(2)'	123.1(2)	As(1)-O(4)-Be(2)	118.2(2)
As(1)-O(4)-Be(2)'	118.2(2)	As(1)-O(5)-Be(2)	127.9(3)

†average bond angle, with standard deviation in [], about central atom.